

Division for Chronological Research



- AMS-¹⁴C dating
- Developing ¹⁴C pre-treatment and measurement techniques
- Analysis of cosmogenic nuclides
- CHIME (chemical U-Th total Pb isochron method)
- Geochronology
- Isotope analysis
- Microanalysis and spectroscopy
- Paleoclimate reconstruction

Short- and long-term forecasts of global environmental changes and their countermeasures are issues of great urgency. Determining when an event occurred in the past, via “dating,” is of importance understanding present and predicting future states of the Earth. We promote chronological studies on a broad range of subjects from events in Earth’s history, spanning ~4.6 billion years, to archaeological materials, cultural properties, and modern cultural assets. The Tandetron dating group conducts interdisciplinary research involving radiocarbon (¹⁴C) dating using accelerator mass spectrometry to understand changes in the Earth’s environment and the cultural history of humankind from ~50,000 years ago to the present day. In addition, the group studies near-future forecasts of Earth and Space environments, focusing on spatiotemporal variations in cosmogenic nuclides, such as ¹⁴C and ¹⁰Be, and conducts research that integrates art and science through collaboration between researchers in archeology, historical science, and other fields. The microscale spatial dating group uses the chemical U-Th total Pb isochron method (CHIME), which was firstly developed at Nagoya University, to shed light on events in Earth’s history from its formation 4.6 billion years ago up to approximately 1 million years ago. An electron probe microanalyzer (EPMA) have been used to perform nondestructive microanalyses of rocks and other materials to reveal records of complex events recorded in zircon, monazite, and other samples.

Main Activities in FY2018

JASPAR – The Japan-Spain-Pakistan Archaeological Research Initiative

JASPAR is a new research initiative in the archaeology of Sindh, the southeast of Pakistan. It is an umbrella of projects dedicated to study the paleoenvironment, archaeology, and ethnoarchaeology of Sindh during the period from early humans (PaleoAsia project) to the Harappan Civilization (2600–1900 BC; RainDrops and ModAgrO projects). It has been promoted by the Shah Abdul Latif University (SALU, Khairpur, Sindh), the Endowment Fund Trust for Preservation of the Heritage of Sindh, the Universitat Pompeu Fabra (UPF, Barcelona, Spain), the Japanese Centre for South Asian Cultural Heritage (JCSACH, Tokyo, Japan), the University of Tokyo, Nagoya University, and the National Institute of Advanced Industrial Science and Technology (AIST). The first international collaborative survey of archaeological and paleoclimate studies in the Thar Desert, Pakistan’s Sindh Province, was carried out from January to February 2018.

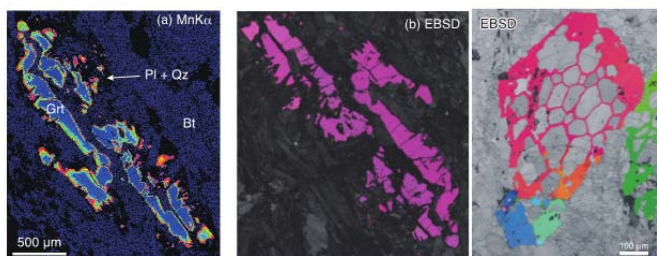


JASPAR survey in the Thar Desert, Pakistan’s Sindh Province.

Analysis of crystallographic orientations of garnet grains in coesite - eclogite

Various scales of deformation structures, such as folding, faulting, pressure shadows, lattice-preferred orientation, and dislocation phenomena, observed in metamorphic rocks and minerals clearly record the dynamic processes during metamorphism. To investigate the behavior of various metamorphic rocks in response to tectonism during prograde and retrograde metamorphic stages, the electron back scatter diffraction (EBSD) method and EPMA were employed in the analysis of crystallographic

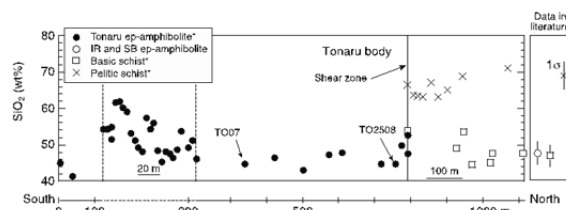
orientations of garnet grains in coesite-eclogite from the Sulu Belt, China; granulite in the Mogok Belt, Myanmar; and epidote-amphibolite and quartz schist of eclogite and non-eclogite units, respectively, in the Sanbagawa Belt, Japan. Most of the analyzed garnet grains showed segmentation texture formed by a hydration reaction during the exhumation stage. The segments that form an aggregate in each sample share similar crystallographic orientations, with misorientations of less than 3–4°. This suggests that the segmentation textures were not formed by deformational crush, but that the grains recorded a static environment during exhumation. A honeycomb garnet, which includes an abundance of quartz grains, in a quartz schist from a Sanbagawa non-eclogite unit also shows no evidence to suggest deformation processes. This grain formed by hydraulic fracturing of quartz and garnet recrystallization along the grain boundaries due to the dehydration reaction during the prograde stage of the epidote-amphibolite facies metamorphism. Thus, this sample might not have undergone significant deformation during exhumation from depths of 25–30 km (0.8–1.0 GPa) to the Earth's surface.



Left and Center: (a) EPMA MnKα and (b) EPSP maps of a segmented garnet in the Mogok granulite from Myanmar. Abbreviations for minerals: Bt, biotite; Grt, garnet; Pl, plagioclase; Qz, quartz.
Right: EBSD map of a honeycomb garnet in a Sanbagawa quartz schist.

Petrological and geochemical studies of Tonaru epidote-amphibolite and surrounding schists in the Sanbagawa Metamorphic Belt, central Shikoku

A subduction zone is a unique environment where various materials from the Earth's surface encounter mafic and ultramafic lithologies of the lower crust and mantle wedge. It can be inferred that mechanical (physical) and chemical interactions between these materials progress dynamically during the subduction process along the interface between the subducted slab and the crust-mantle zone beneath the arc-trench system. In the Besshi region of the Sanbagawa Metamorphic Belt in central Shikoku, SW Japan, there are extensive occurrences of various metamorphic rocks that have originated from different protoliths of peridotite, gabbro, basalt, shale-sandstone, and limestone. Field relationships between the Tonaru epidote-amphibolite body and the surrounding pelitic and basic schists are well-exposed along the Kokuryo River in the Tonaru area of the Sanbagawa Belt in the Besshi region. Layers and lenses of marble and pelitic schist occur in the southern part of the Tonaru body. However, the petrological and geochemical characteristics of these lithologies and their origins have not yet been elucidated in detail. The petrographic characteristics and variations in whole-rock compositions of the Tonaru epidote-amphibolite suggest that the Tonaru epidote-amphibolite is a metamorphosed composite body of layered gabbro and gabbro-sediment mixtures composed of mafic, ultramafic, and/or pelitic materials, likely derived from an oceanic island arc.



Variation of SiO₂ concentrations (wt%) in the Tonaru epidote-amphibolites along the Kokuryo River.

Drilling of marine sediment in the Amundsen Sea to detect melting events of West Antarctic ice sheet

A sector of the West Antarctic ice sheet draining into the Amundsen Sea is undergoing the largest ice loss in Antarctica today, and there is serious concern for the potential of large-scale ice collapse of this area due to global warming. The International Ocean Discovery Program (IODP) Expedition 379 drilled two sites in the Amundsen Sea area of the Southern Ocean using the D/V JOIDES Resolution, in January to March 2019. A researcher from ISEE participated in this expedition and measured the physical

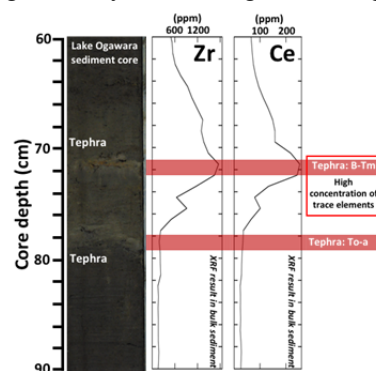


Photo: D/V JOIDES Resolution.

properties of marine sediment cores. It is expected that melting events of the West Antarctic ice sheet during the Plio-Pleistocene will be detected by future research.

Chemical characterization of B-Tm tephra (Millennium Eruption, Changbaishan volcano) of a lacustrine sediment core

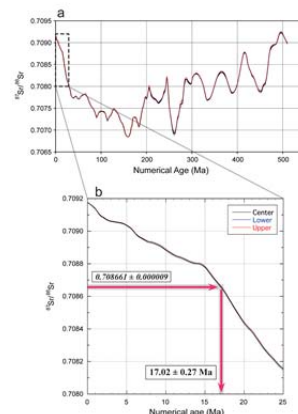
The absolute date of the Millennium Eruption (ME) from the Changbaishan volcano (also referred to as Mt. Paektu, Baegdusan, or Tianchi), located at the border between China and North Korea (128°03'E, 41°00'N), has been refined to 946 AD. This indicates that tephrochronology using widely dispersed B-Tm tephra deposits has great utility in obtaining a robust age constraint in the late Holocene period. Here, we present multiple geochemical datasets, such as those obtained by X-ray fluorescence, solution inductively coupled plasma-mass spectrometry, and laser ablation inductively coupled plasma-mass spectrometry, as well as EPMA, of bulk sediments and individual glass shard samples from the B-Tm tephra deposit layer retrieved from Lake Ogawara, Japan. Our results show that the B-Tm tephra layer has extremely high amounts of trace elements (e.g., Zr and Ce for 1,700 ppm and 230 ppm, respectively, in bulk sediments), which is comparable to the proximal B-Tm tephra deposit. Furthermore, the B-Tm tephra layer shows a distinctive chemical composition compared to those of other Japanese tephra. These results indicate that measurements of trace elements, including rare earth elements, have great utility in the identification and correlation of the B-Tm layer.



Depth profile of Zr and Ce in a core of Lake Ogawara sediment alongside a photograph of the core.

Accurate age determination using Sr isotope ratios of rapidly formed spherical carbonate concretions

Spherical carbonate (CaCO_3) concretions often occur in finer-grained marine sediments of varying geological ages. Recent studies have revealed that they form very rapidly under tightly constrained conditions. However, the formation ages of isolated spherical carbonate concretions have never been determined. Therefore, we used $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of isolated spherical carbonate concretions to determine the formation ages. The strontium isotopic stratigraphy obtained using $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of all concretions indicate age determinations with higher accuracy than those estimated using micro-fossils. The results imply that the $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of isolated spherical carbonate concretions can be applied to determine the precise numerical age of marine sediments when the concretions form soon after sedimentation. The age determinations have high accuracy in cases even without any fossil evidence.



Sr isotopic stratigraphy and numerical age determinations from concretions (Yoshida et al., 2019)

Evaluation of matrix correction procedure in quantitative EPMA

The accuracy of quantitative EPMA of U, Th, and Pb is an essential factor in obtaining a reliable age of an analyzed mineral. Quantitative EPMA estimates chemical compositions from X-ray intensities of standard materials and unknown target, and those of standard materials through the matrix correction procedure. The accuracy of matrix correction depends on the chosen models and physical parameters, such as mass attenuation coefficients. CHIME ages of two standard monazites have been determined with various matrix correction models and physical parameters to evaluate the reliability of each model and parameter. A comparison of CHIME and isotopic ages shows that PAP, XPhi, and Bence-Albee method with correction factors by Kato (2005) give an accurate CHIME age, while other models give systematically older ages than isotopic ages.

Comparison of CHIME and LA-ICP-MS ages

Skrzypek et al. (2018) analyzed monazite grains with both LA-ICP-MS and EPMA-CHIME. EPMA-CHIME dating gives smaller uncertainty and variation between samples than LA-ICP-MS U-Pb dating. However, EPMA-CHIME dating gives systematically younger ages than LA-ICP-MS dating. The result implies that EPMA-CHIME dating is sensitive to the difference between U-Pb and Th-Pb systems and the Th-Pb system is sensitive to the effect of retrograde metamorphism.

^{14}C dating of various carbon components in ground ice in Siberia

Recently, the ground ice in permafrost has received attention as a useful tool for paleoenvironment reconstruction. It is important to determine the formation age of ground ice to understand past climate changes and hydrological environmental changes preserved in the ground ice. To define which carbon component is most suitable for the determination of the formation ages of ground ice, we have measured ^{14}C ages of various components such as particulate organic carbon (POC), dissolved organic carbon (DOC), dissolved inorganic carbon (DIC), and CO_2 gas in bubbles in ground ice from the outcrops of permafrost in Syrdakh and Churapcha, near Yakutsk City in Russia. The ^{14}C ages of POC in the ground ice samples were $\sim 10,000$ years older than ages of the plant remain, indicating that POC is not suitable for use in determining the age of ground ice formation. Meanwhile, the DIC and bubble CO_2 ages were $\sim 10,000$ years younger than the plant ages. The ^{14}C ages of DOC were different by molecular size: the $0.7\ \mu\text{m}$ -10 kDa and 10 kDa-3 kDa fractions showed similar ages to those of plant remains, whereas the $<3\ \text{kDa}$ fraction showed younger ages, which were similar to those of DIC and bubble CO_2 . The results obtained in this study led to the elucidation of formation processes of ground ice and paleoenvironmental reconstruction.



Outcrop of permafrost in Churapcha.

^{14}C ages of wood blocks from volcanic mudflow deposits: Examples for Maebashi and Tsukahara deposits

Volcanic debris-flow avalanches and mudflows caused by the collapse of volcanic edifices are gravity currents that involve surface materials, including water, plants, rocks, and soils derived from stream sediments and near-surface deposits. Wood blocks are useful materials for ^{14}C dating to determine the age of events, especially hazardous events. However, reworked fragments from deposits of preceding events might provide older ages. Therefore, careful sampling and measurement of wood specimens are required for a correct evaluation of the history of volcanic edifices. We performed ^{14}C measurements for wood specimens from the Maebashi and Tsukahara mudflow deposits in central Japan. The results suggested that some collapses of the Asama volcano, although of unknown scale, occurred more than 10,000 years before and after the main event of $\sim 27,000$ years ago.



Expanded region of the Maebashi and Tsukahara mudflows.

Radiocarbon dating of peach stones from Makimuku ruins, Nara Prefecture

High-precision radiocarbon dating was performed on 12 aliquots of 2,800 peach stones excavated at the Makimuku ruins, Nara Prefecture, Japan. The peaches were ripened, eaten, and disposed of during a period of ~ 100 years from 135 AD to 230 AD. This confirms that the Makimuku ruins is one of the more convincing candidate sites supporting the theory that Yamataikoku was located in Kinai region.



Remnant peach stone excavated from the Makimuku archeological site.